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4 Jun 1980(51) INT CL³ C09B 45/28(52) Domestic classification
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8B2 8B3 8D1

(56) Documents cited

GB 1094956

GB 1094343

GB 1091454

GB 923265

GB 864276

(58) Field of search
C4P(71) Applicant
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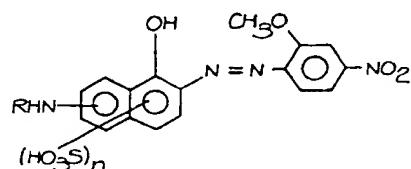
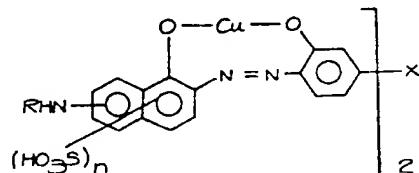
Schündehütte

Dietrich Grabman

(74) Agents
Carpmaels & Ransford(54) Process for the preparation of
copper-containing azo dyestuffs

formula II

(57) Dyestuffs of the formula



are reacted with a copper donating agent—preferably in the presence of 0.5–10% ammonia—and the nitro group of the reaction products are reduced in the usual manner.

wherein

R = H, acetyl, benzoyl or phenyl

n = 1 or 2

x = azo or azoxy bridge

are obtained more economically as
compared with processes of the
prior art if compounds of the for-

ERRATUM

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Front page heading (72) Inventors for Dietrich Grabman read Dietrich Graßman

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5 September 1980

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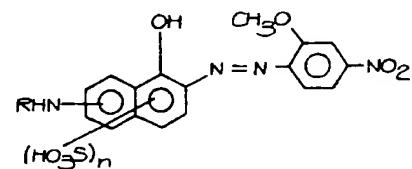
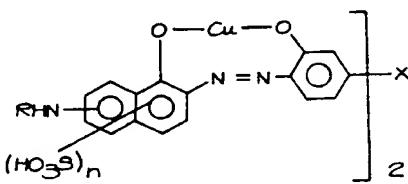
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(54) Process for the preparation of
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of 0.5–10% ammonia—and the ni-
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wherein

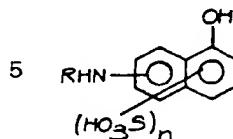
R = H, acetyl, benzoyl or phenyl

n = 1 or 2

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are obtained more economically as
compared with processes of the
prior art if compounds of the for-

GB 2 034 344 A



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in which

R and n have the abovementioned meaning, for example to 1-hydroxy-8-aminonaphthalene-3,6-disulphonic acid, 1-hydroxy-8-acetylaminonaphthalene-3,6-disulphonic acid, 4-hydroxy-7-aminonaphthalene-2-sulphonic acid, 4-hydroxy-7-acetylaminonaphthalene-2-sulphonic acid, 4-hydroxy-7-benzoylaminonaphthalene-2-sulphonic acid, 4-hydroxy-7-phenylaminonaphthalene-2-sulphonic acid, 4-hydroxy-6-aminonaphthalene-2-sulphonic acid, 4-hydroxy-6-acetylaminonaphthalene-2-sulphonic acid, 4-hydroxy-6-benzoylaminonaphthalene-2-sulphonic acid, 4-hydroxy-6-phenylaminonaphthalene-2-sulphonic acid, 4-hydroxy-6-(3'-carboxyphenylamino)-naphthalene-2-sulphonic acid, 4-hydroxy-6-(4'-carboxyphenylamino)-naphthalene-2-sulphonic acid, 4-hydroxy-6-(3'-sulphophenylamino)-naphthalene-2-sulphonic acid and 4-hydroxy-6-(4'-sulphophenylamino)-naphthalene-2-sulphonic acid.

Surprisingly, a higher profitability is achieved with the process according to the invention than with one of the processes described in German Patent Specifications 636,358 or 694,965 or in German Auslegeschrift 1,544,559.

40 In the examples which follow, "parts" denote parts by weight, "percentages" denote percentages by weight and the temperatures are given in degrees Centigrade.

45 EXAMPLE 1:

41.7 parts of the nitroazo compound 3-(4'-nitro-2'-methoxyphenylazo)-4-hydroxy-6-(3'-sulphophenylamino)-naphthalene-2-sulphonic acid are suspended in 600 parts of water, the suspension is heated to 80°C and a solution of 20 parts of copper-II sulphate pentahydrate in 50 parts of water and 50 parts of an aqueous ammonia solution which contains 25 per cent of ammonia are added. The mixture is heated to 95-98°C and stirred at this temperature for four hours, during which the copper complex of the nitroazo compound dissolves. When the reaction has ended, the product is precipitated by adding 200 parts of sodium chloride and is isolated. The resulting moist paste is suspended in 750 parts of water, the suspension is heated to 90° and 50 parts of an aqueous sodium hydroxide solution which contains 30 per cent of sodium hydroxide are added. A solution of 10 parts of

glucose in 50 parts of water is now added in the course of five minutes. Immediately thereafter, the dyestuff is precipitated by adding 150 parts of sodium chloride and is isolated.

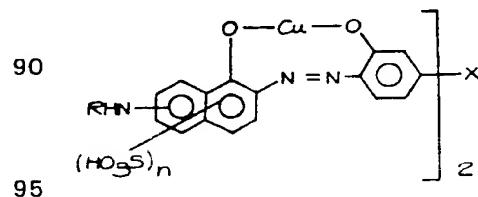
70 After drying, 37.6 parts of a black-blue powder which dyes cotton in blue-grey shades are obtained.

EXAMPLE 2:

75 The same dyestuff is obtained if, instead of the 41.7 parts of the nitroazo compound from Example 1, an amount equivalent thereto of the suspension obtained on coupling diazotised 5-nitro-2-aminoanisole to 4-hydroxy-6-80 (3'-sulphophenylamino)-naphthalene-2-sulphonic acid and 40 parts of copper-II sulphate pentahydrate are used.

CLAIMS

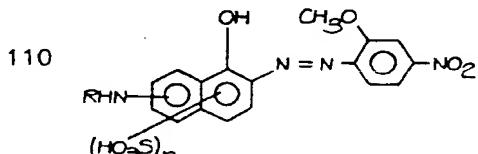
85 1. A process for the production of a dyestuff of the general formula



in which

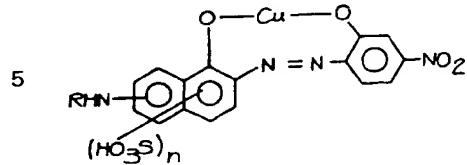
100 R denotes a hydrogen atom or an acetyl, benzoyl or optionally further substituted phenyl radical,
n is 1 or 2 and
X denotes an azo and/or azoxy bridge.

105 in which a compound of the general formula



in which

120 R and n have the above-mentioned meanings, is treated with an agent which donates copper and is thereby converted into a compound of the general formula



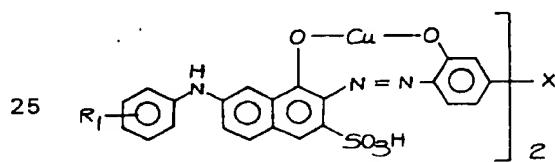
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in which

R and n have the above-mentioned meanings, and the compound of formula (III) is then reduced.

2. A process according to claim 1 for the production of a compound of the general formula

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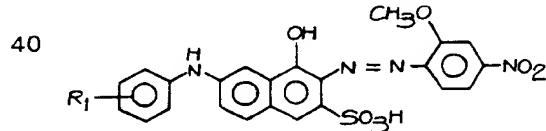


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in which

R₁ denotes 3-SO₃H, 4-SO₃H, 3-COOH or 4-COOH and

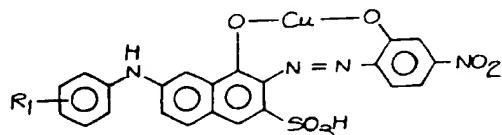
35 X has the same meanings as in claim 1, in which a compound of the general formula



45

in which

50 R₁ has the above-mentioned meaning, is treated with an agent which donates copper and is thereby converted into a compound of the general formula



in which

75 R₁ has the above-mentioned meaning, and the compounds of formula (VI) is then reduced.

3. A process according to claim 1 or 2, in which the treatment with an agent which donates copper is carried out in the presence of 0.5 to 10% by weight of ammonia.

4. A process according to claim 1 when carried out substantially as described in Example 1 or 2.

80 5. A dyestuff of formula (I) as defined in claim 1 when produced by the process of any of the foregoing claims.

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